




## ACIDIC OIL-IN-WATER TYPE EMULSION COMPOSITION

**Patent number:** JP2002176952  
**Publication date:** 2002-06-25  
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**Classification:**  
**- international:** A23D7/01; A23L1/24; A23L1/32;  
A61K9/107; A61K47/14; A23D7/005;  
A23L1/24; A23L1/32; A61K9/107;  
A61K47/14; (IPC1-7): A23L1/24  
**- european:** A23D7/01N; A23L1/24; A23L1/32;  
A61K9/107; A61K47/14  
**Application number:** JP20000381596 20001215  
**Priority number(s):** JP20000381596 20001215

**Also published as:**

 EP1214886 (A1)  
 US6743459 (B2)  
 US2002119239 (A1)

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**Abstract of JP2002176952**

**PROBLEM TO BE SOLVED:** To obtain an acidic oil-in-water type emulsion composition having excellent preservation stability at low temperatures, a good appearance and a good flavor and useful as a food for dieting and a food for improving lipid metabolism though a diacylglycerol(DAG) is contained at a high concentration. **SOLUTION:** This acidic oil-in-water type emulsion composition comprises  $\geq 20$  wt.% of the DAG and an oily phase containing 0.5-5.0 wt.% of a crystal inhibitor.

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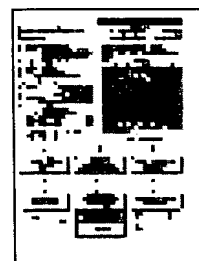
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Kind: **A2 Document Laid open to Public inspection**Inventor: **SHIIBA DAISUKE;  
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[News, Profiles, Stocks and More about this company](#)Published /  
Filed: **2002-06-25 / 2000-12-15**Application  
Number: **JP2000000381596**IPC Code: **A23L 1/24;**Priority  
Number: **2000-12-15 JP2000000381596**Abstract: **PROBLEM TO BE SOLVED:** To obtain an acidic oil-in-water type emulsion composition having excellent preservation stability at low temperatures, a good appearance and a good flavor and useful as a food for dieting and a food for improving lipid metabolism though a diacylglycerol(DAG) is contained at a high concentration.**SOLUTION:** This acidic oil-in-water type emulsion composition comprises  $\geq 20$  wt.% of the DAG and an oily phase containing 0.5-5.0 wt.% of a crystal inhibitor.**COPYRIGHT: (C)2002,JPO**View  
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PT SE TR**Family: [Show 4 known family members](#)Other: **CHEMABS 137(01)005449K**

Abstract Info:

(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開2002-176952

(P2002-176952A)

(43) 公開日 平成14年6月25日 (2002.6.25)

(51) Int.Cl.<sup>7</sup>

識別記号

F I

テ-マコ-ト\* (参考)

A 2 3 L 1/24

A 2 3 L 1/24

A 4 B 0 4 7

審査請求 未請求 請求項の数 5 O L (全 5 頁)

(21) 出願番号 特願2000-381596 (P2000-381596)

(22) 出願日 平成12年12月15日 (2000.12.15)

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(54) 【発明の名称】 酸性水中油型乳化組成物

(57) 【要約】

【課題】 高濃度のDAGを含有するにもかかわらず、低温での保存安定性に優れ、かつ外観や風味も良好で、ダイエット用食品や脂質代謝改善食品として有用な酸性水中油型乳化組成物の提供。

【解決手段】 ジアシルグリセロール20重量%以上及び結晶抑制剤0.5~5.0重量%を含む油相を含有する酸性水中油型乳化組成物。

## 【特許請求の範囲】

【請求項1】 ジアシलगリセロール20重量%以上及び結晶抑制剤0.5~5.0重量%を含む油相を含有する酸性水中油型乳化組成物。

【請求項2】 結晶抑制剤が、ポリグリセリン脂肪酸エステル、ショ糖脂肪酸エステル及びソルビタン脂肪酸エステルから選ばれるものである請求項1記載の酸性水中油型乳化組成物。

【請求項3】 ポリグリセリン脂肪酸エステルが、平均重合度2~12、構成脂肪酸の炭素数12~22、エステル化率70%以上のものである請求項2記載の酸性水中油型乳化組成物。

【請求項4】 ショ糖脂肪酸エステルが、炭素数12~22の脂肪酸によるエステル化率が50%以上であり、かつ、当該脂肪酸によりエステル化されていない水酸基がアセチル化されているものである請求項2記載の酸性水中油型乳化組成物。

【請求項5】 ソルビタン脂肪酸エステルが、構成脂肪酸の炭素数12~22、HLB3未満のものである請求項2記載の酸性水中油型乳化組成物。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、冷蔵庫内等の低温条件下でも乳化安定性に優れ、かつ外観、風味も良好であり、ダイエット用食品や脂質代謝改善食品として有用なマヨネーズ・ドレッシング等の酸性水中油型乳化組成物に関する。

## 【0002】

【従来の技術】近年、ジアシलगリセロール（以下「DAG」という）が、肥満防止作用、体重増加抑制作用等を有することが明らかにされるに至り（特開平4-300828号公報等）、これを各種食品に配合する試みがなされている（特許第2848849号等）。

【0003】しかし、特に、マヨネーズ、ドレッシング等、酸性水中油型乳化組成物の場合、冷蔵庫内条件下（-5~5℃）においては、原料油中のDAGの一部が結晶化して乳化破壊（オイルオフ）を生じ、特にマヨネーズの場合には亀裂が発生するなど、低温耐性が不十分であった。

## 【0004】

【発明が解決しようとする課題】したがって本発明は、高濃度のDAGを含有するにもかかわらず、低温での保存安定性に優れるとともに、外観や風味も良好で、ダイエット用食品や脂質代謝改善食品として有用な酸性水中油型乳化組成物を提供することを目的とする。

## 【0005】

【課題を解決するための手段】本発明者らは、DAGを含む油相中に特定量の結晶抑制剤を含有させることにより、上記要求を満たす酸性水中油型乳化組成物が得られることを見出した。

【0006】すなわち本発明は、ジアシलगリセロール20重量%以上及び結晶抑制剤0.5~5.0重量%を含む油相を含有する酸性水中油型乳化組成物を提供するものである。

## 【0007】

【発明実施の形態】本発明で使用するDAGは、グリセリンの1位と2位又は1位と3位の水酸基が脂肪酸でエステル化されたものである。脂肪酸残基の炭素数に特に制限はないが、8~24、特に16~22が好ましい。また不飽和脂肪酸残基の量が、全脂肪酸残基の55%以上であることが好ましく、更には70%以上、特に90%以上が好ましい。DAGは、植物油、動物油等とグリセリンとのエステル交換反応、又は上記油脂由来の脂肪酸組成物とグリセリンとのエステル化反応等、任意の方法により得られる。反応方法は、アルカリ触媒等を用いた化学反応法、リパーゼ等の油脂加水分解酵素を用いた生化学反応法のいずれでもよい。DAGは1種以上を使用でき、油相中の含有量は、ダイエット用食品としての有効性及び経済性の観点から、20重量%以上であることが必要であり、35重量%以上が好ましい。油相は、DAG以外に、トリアシलगリセロール、モノアシलगリセロール、遊離脂肪酸等を含有していてもよい。

【0008】本発明で使用する結晶抑制剤としては、ポリグリセリン脂肪酸エステル、ショ糖脂肪酸エステル、ソルビタン脂肪酸エステルから選ばれるものが好ましい。ポリグリセリン脂肪酸エステルとしては、グリセリンの平均重合度が2~12、構成脂肪酸の炭素数12~22、エステル化率70%以上のものが好ましく、更にHLBが4.5未満、特に3.5未満のものが好ましい。ショ糖脂肪酸エステルとしては、炭素数12~22の脂肪酸によるエステル化率が50%以上であり、かつ、当該脂肪酸によりエステル化されていない水酸基がアセチル化されているものが好ましく、更にHLBが3未満、特に2未満のものが好ましい。ソルビタン脂肪酸エステルとしては、構成脂肪酸の炭素数12~22、HLBが3未満、特に2.5未満のものが好ましい。結晶抑制剤は1種以上を使用でき、油相中の含有量は、低温での十分な結晶抑制効果及び食品としての風味の観点から0.5~5.0重量%であることが必要であり、0.6~3.0重量%が好ましい。

【0009】本発明の酸性水中油型乳化組成物の水相には、水；酢；食塩；グルタミン酸ナトリウム等の調味料；砂糖、水飴等の糖類；酒、みりん等の呈味量；各種ビタミン；有機酸；香辛料；各種野菜又は果実；キサンタンガム等の増粘剤；牛乳等の乳製品；各種果汁類；大豆タンパク質等のタンパク質類；各種リン酸塩等を含有させることができる。

【0010】本発明の酸性水中油型乳化組成物における油相と水相の配合比（重量比）は、10：90~80：20、特に50：50~75：25が好ましい。

【0011】本発明の酸性水中油型乳化組成物には、乳

化性付与と風味向上のため、卵黄を使用することができ、卵黄は、生、凍結、粉末、加塩、加糖等、任意の形態で使用することができ、また卵白を含んだ全卵の形態で使用してもよい。本発明の酸性水中油型乳化組成物中の卵黄の含有量は、風味向上の観点から、液状卵黄換算で5～20重量%が好ましく、更には7～17重量%、特に8～15重量%が好ましい。

【0012】本発明の酸性水中油型乳化組成物は、保存安定性、外観、風味の観点から、含有する全リン脂質に対するリゾリン脂質の比率（以下、「リゾ比率」という）が15%以上であることが好ましく、更には25%以上、特に29～60%であることが好ましい。リゾリン脂質は、その一部又は全部が卵黄や大豆由来であることが好ましく、卵黄由来であることが特に好ましい。また、リゾリン脂質の一部又は全部が酵素処理卵黄であることが好ましい。卵黄の酵素処理に用いる酵素としては、エステラーゼ、リパーゼ、ホスホリパーゼが好ましく、リパーゼ、ホスホリパーゼがより好ましく、ホスホリパーゼが特に好ましい。ホスホリパーゼの中でも、ホスホリパーゼA、すなわちホスホリパーゼA1及びA2が最も好ましい。

【0013】本発明の酸性水中油型乳化組成物の製品形態としては、例えば日本農林規格（JAS）で定義されるドレッシング、半固体状ドレッシング、乳化液状ドレッシング、マヨネーズ、サラダドレッシング、フレンチドレッシング等が挙げられるが、特にこれらに限定されるものではなく、広くマヨネーズ類、ドレッシング類といわれるものが該当する。

【0014】本発明の酸性水中油型乳化組成物は、例えば以下の方法により製造することができる。まず、DA G、結晶抑制剤等の油性成分を混合して油相を調製する。また、卵黄、その他水溶性原料を混合して水相を調製する。該水相に該油相を添加し、必要により予備乳化を行い、均質化することにより、酸性水中油型乳化組成物を得ることができる。均質機としては、例えばマウンテンゴウリン、マイクロフルイダイザー等の高圧ホモジナイザー、超音波式乳化機、コロイドミル、アジホモミキサー、マイルダー等が挙げられる。

【0015】本発明の酸性水中油型乳化組成物は、通常のマヨネーズ、ドレッシング等と同様に使用することができる。

【0016】

【実施例】以下において、結晶抑制剤のHLBは、ポリグリセリン脂肪酸エステル及びソルビタン脂肪酸エステルについてはGriffinの経験式、ショ糖脂肪酸エステルについては乳化法により算出した。

【0017】参考例1 油脂組成物1の調製

ナタネ油脂肪酸650重量部とグリセリン107重量部の混合物に、リボザイムIM（ノボ・ノルディスクインダストリー社製）を加え、40℃、5時間、7hPaでエステル化反

応を行った後、分子蒸留（235℃、0.07hPa）を行った。次いで脱色、水洗し、235℃で2時間脱臭し、表1に示す組成の油脂組成物1を得た。

【0018】参考例2 油脂組成物2の調製

ウィンタリングして飽和脂肪酸含量を少なくしたダイズ油脂肪酸650重量部、グリセリン107重量部及び水酸化カルシウム2重量部の混合物を、窒素ガス雰囲気下で230℃で0.5時間反応を行った後、12時間静置し、グリセリン相を除去してから、油相（油脂組成物）100重量部に対して2重量部の50重量%クエン酸水溶液で水洗し、遠心分離法で油脂組成物を取り出した。次いで分子蒸留（235℃、0.07hPa）し、脱色、水洗し、235℃で2時間脱臭し、表1に示す組成の油脂組成物2を得た。

【0019】

【表1】

		油脂組成物	
		1	2
油脂組成*1	トリアシルグリセロール	13.8	13.5
	ジアシルグリセロール	84.7	85.2
	モノアシルグリセロール	1.2	1.0
	遊離脂肪酸	0.3	0.3
構成脂肪酸*2	C16:0	4.2	2.6
	C18:0	1.9	0.7
	C18:1	58.3	30.0
	C18:2	21.3	57.5
	C18:3	10.7	6.8
	C20:0	0.7	1.5
	C20:1	1.8	0.4
	C22:0	0.2	0.1
	C22:1	0.8	0.1

\*1:シリル化後、ガスクロマトグラフィーで分析

\*2:メチル化後、ガスクロマトグラフィーで分析  
（脂肪酸の炭素数：炭素-炭素二重結合の数）

【0020】参考例3 酵素処理卵黄の調製1

食塩濃度10重量%の卵黄液750g及び水250gを混合し、50℃で10分予熱した後、卵黄液に対して100ppmのホスホリパーゼA2（酵素活性10,000IU/ml）を添加し、3～5時間反応を行い酵素分解卵黄液を得た。なお、リゾ化率は以下の方法により算出した。

【0021】まず反応物をクロロホルム：メタノールの混合溶媒（3：1）により繰り返し抽出を行い、脂質混合物を得た。得られた脂質混合物を、薄層クロマトグラフィーに供し、一次元：クロロホルム-メタノール-水（65：25：49）、二次元：ブタノール-酢酸-水（60：20：20）による二次元薄層クロマトグラフィーにより、各種のリン脂質を分取した。分取した各種のリン脂質量は、市販測定キット（過マンガン酸塩灰化法、リン脂質-テストワコー；和光純薬工業株式会社）を用いて測定を行い、リゾ化リン脂質、全リン脂質含量を算出した。リゾ化率（%）は、『（リゾ化リン脂質画分リン合計量／全リン脂質画分リン合計量）×100』により算出した。

## 【0022】参考例4 酵素処理卵黄の調製2

参考例3で得られた酵素分解卵黄液に、2倍容の水を加え攪拌後、入口空気温度170℃、出口空気温度70℃にて噴霧乾燥を行い、酵素処理卵黄粉末を得た。

## 【0023】試験例1（実施例1～8及び比較例1～3）

常法に従い表2に示す組成の油相及び水相を調製した。水相を攪拌しながら油相を添加し、予備乳化した後、コロイドミル（5000rpm、クリアランス0.35mm）で均質化し、平均乳化粒子径2.5～3.5μmのマヨネーズを製造し、100gマヨネーズチューブに充填した。

【0024】得られた各マヨネーズを、-5～5℃で1カ月保存した後、約3時間室温に放置し、その外観及び物性を、6名のパネラーにより、以下の評価基準に従って\*

\*で評価した。この評価の平均値を表2に示す。

## 【0025】◇評価基準

(1) 外観・・・チューブ入りマヨネーズの外観を目視評価した。

3：良好

2：一部分離が認められる

1：著しい分離が認められる

【0026】(2) 物性・・・チューブから絞り出したマヨネーズを目視評価した。

3：良好

2：一部肌荒れ、離水等が認められる

1：著しい肌荒れ、離水等が認められる

## 【0027】

## 【表2】

(重量%)

		実施例								比較例		
		1	2	3	4	5	6	7	8	1	2	3
水相	食塩	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30
	上白糖	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	調味料	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
	からし粉	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
	増粘剤 <sup>*1</sup>	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
	酵素処理卵黄	20.00	20.00	20.00	20.00	20.00	20.00	—	—	20.00	20.00	20.00
	酵素処理粉末卵黄	—	—	—	—	—	—	7.40	3.70	—	—	—
	卵黄	—	—	—	—	—	—	—	7.50	—	—	—
油相	10%醸造酢	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00
	水	1.90	1.90	1.90	1.90	1.90	1.90	14.50	10.70	1.90	1.90	1.90
	油脂組成物1	20.7	20.5	19.5	20.5	20.5	34.5	20.5	20.5	21.0	35.0	20.8
	油脂組成物2	48.7	48.5	47.5	48.5	48.5	34.5	48.5	48.5	49.0	35.0	48.8
リゾ化率 (%)	ポリグリセリン脂肪酸エステル <sup>*2</sup>	0.60	1.00	3.00	—	—	1.00	1.00	1.00	—	—	0.40
	ソルビタン脂肪酸エステル <sup>*3</sup>	—	—	—	1.00	—	—	—	—	—	—	—
	ショ糖脂肪酸エステル <sup>*4</sup>	—	—	—	—	1.00	—	—	—	—	—	—
	水	—	—	—	—	—	—	—	—	—	—	—
評価	5℃	外観	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
	5℃	物性	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
	0℃	外観	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	2.0	3.0
	0℃	物性	3.0	3.0	3.0	3.0	3.0	3.0	3.0	2.5	1.5	3.0
	-5℃	外観	3.0	3.0	3.0	3.0	3.0	3.0	3.0	1.0	1.0	1.0
	-5℃	物性	3.0	3.0	3.0	3.0	3.0	3.0	3.0	1.0	1.0	1.0

## 【0028】

\*1：キサンタンガム（大日本製薬社製）

\*2：平均重合度：10，エステル化率：80%以上，構成脂肪酸：C18:1及びC16，HLB=2.0

\*3：構成脂肪酸：C18，HLB=2.1

\*4：構成脂肪酸：C18及びC2，C18エステル化率：60%以上，アセチル化率：35%以上，HLB=0

【0029】比較例1及び2は、結晶抑制剤を配合していないマヨネーズであり、それぞれ-5℃及び0℃で1ヶ月間の保存で乳化破壊（オイルオフ）し、分離や肌荒れ、離水が発生した。比較例3は、油相中にポリグリセリン脂肪酸エステルを0.4重量%使用したものであり、充分な低温乳化安定性が得られなかった。

【0030】これに対し、結晶抑制剤として、ポリグリセリン脂肪酸エステル（実施例1～3、6～8）、ソルビタン脂肪酸エステル（実施例4）又はショ糖脂肪酸エ

ステル（実施例5）を適正量使用した場合、-5～5℃で1ヶ月間の保存でも乳化破壊は発生せず、外観、物性ともに良好なものであった。

## 【0031】試験例2（実施例9～11）

表3に示す割合でフレンチドレッシング（実施例9）、サウザンドアイランドドレッシング（実施例10）及びごまドレッシング（実施例11）を製造した。すなわち、油相原料を水相に攪拌下滴下し、予備乳化を行った。これをホモミキサーにより均質化し、平均乳化粒子径4～12μmの各ドレッシングを得た。各ドレッシングについて、試験例1と同様に6名のパネラーにより評価を行った結果、表3に示すように、いずれも-5～5℃で1ヶ月間の保存後も、外観、物性ともに良好であった。

## 【0032】

## 【表3】

			実施例			
			9	10	11	
水相	食塩		3.00	2.00	2.50	
	上白糖		5.00	5.00	11.00	
	調味料		0.50	0.50	1.00	
	レモン果汁		2.00	2.00	—	
	増粘剤*1		0.60	0.40	0.01	
	酵素処理卵黄		2.00	4.00	—	
	卵黄		—	—	1.50	
	トマトケチャップ		—	5.00	—	
	ピクルス		—	4.00	—	
	トマトペースト		—	1.00	—	
	みそ		—	—	4.00	
	すりごま		—	—	7.00	
	しょう油		—	—	3.00	
油相	5%醸造酢		14.00	14.00	14.00	
	水		32.90	27.10	25.99	
	油脂組成物 1		19.5	17.0	14.5	
	油脂組成物 2		19.5	17.0	14.5	
	ポリグリセリン脂肪酸エステル*2		1.00	1.00	1.00	
	リソ化率 (%)		40	40	—	
評価	5℃	外観		3.0	3.0	3.0
		物性		3.0	3.0	3.0
	0℃	外観		3.0	3.0	3.0
		物性		3.0	3.0	3.0
	-5℃	外観		3.0	3.0	3.0
		物性		3.0	3.0	3.0

【0033】

\*1: キサンタンガム (大日本製薬社製)

\*2: 平均重合度: 10, エステル化率: 80%以上, 構成  
脂肪酸: C18:1及びC16

【0034】

【発明の効果】本発明の酸性水中油型乳化組成物は、DA  
Gを高濃度に含有するにもかかわらず、冷蔵庫内での低  
温保存中に亀裂が発生することがないなど低温での保存  
安定性に優れ、かつ外観及び風味も良好であり、ダイエ  
ット用食品や脂質代謝改善食品として有用である。

フロントページの続き

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Fターム(参考) 4B047 LB09 LG06 LG08 LG66

## ACIDIC OIL-IN-WATER TYPE EMULSION COMPOSITION

Description of corresponding document: **EP1214886**

### BACKGROUND OF THE INVENTION

Field of the Invention:

[0001] The present invention relates to an acidic oil-in-water type emulsion composition such as mayonnaise or dressing, which is excellent in emulsion stability even under low-temperature conditions such as in side refrigerators, good in appearance and flavor and useful as diet or food for improving lipid metabolism.

Description of the Background Art:

[0002] In recent years, it has been clarified that diacylglycerol (hereafter referred to as "DAG") has an obesity-preventing effect, an effect to prevent increase in weight, etc. (Japanese Patent Application Laid-Open No. 300828/1992, etc.), and it is attempted to incorporate this into various kinds of foods (Japanese Patent Application Laid-Open Nos. 8431/1991, 79858/1992 and 176181/1998, etc.). Furthermore, oil-in water type emulsions containing DAG and lecithin in an oil phase and protein in a water phase have been proposed (Japanese Patent Application Laid-Open No. 91451/1991)

[0003] In the case of, particularly, an acidic oil-in-water type emulsion composition such as mayonnaise or dressing, however, the low-temperature resistance has been insufficient in that a part of DAG in raw oil crystallizes under in-refrigerator conditions (-5 to 5 DEG C) that cause demulsification (oil-off), and for mayonnaise, cracks were observed.

### SUMMARY OF THE INVENTION

[0004] It is therefore an object of the present invention to provide an acidic oil-in-water type emulsion composition which has excellent shelf stability at low temperatures though containing DAG at a high concentration, also good in appearance and flavor and useful as a diet or food for improving lipid metabolism.

[0005] The present inventors have found that an acidic oil-in-water type emulsion composition satisfying the above requirements can be provided by containing a specified amount of crystallization inhibitors in an oil phase containing DAG.

[0006] Thus, according to the present invention, we inventors have provided an acidic oil-in-water type emulsion composition comprising an oil phase containing at least 20 % by weight of diacylglycerol and 0.5 to 5.0 % by weight of a crystallization inhibitor.



## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0007] DAG used in the present invention is obtained by esterifying hydroxyl groups at 1- and 2-positions or 1-and 3-positions of glycerol with fatty acids. The number of carbon atoms in fatty acid residues are preferably (but not limited to) 8 to 24, particularly 16 to 22. The amount of unsaturated fatty acid residues is preferably at least 55%, more preferably at least 70%, most preferably at least 90% based on all the fatty acid residue. DAG is obtained by any known process such as ester exchange reaction of vegetable oil and/or animal oil with glycerol or esterification reaction of fatty acids derived from the above oil with glycerol. The reaction method thereof may be either a chemical reaction by the aid of alkali catalysts, for example, or a biochemical reaction method using oil and/or fat hydrolase such as lipase. One or blends of such DAGs may be used. The content of DAG in the oil phase must be at least 20 % by weight, preferably at least 35 % by weight from the viewpoints of effectiveness as a diet and profitability. The oil phase may contain triacylglycerol, monoacylglycerol, fatty acids and the like in addition to DAG. These may be derived from any of vegetable oils and/or animal oils.

[0008] Examples of raw oil include vegetable oils such as soybean oil, rapeseed oil, sunflower oil, cotton-seed oil, corn oil, safflower oil, linseed oil, olive oil, rice oil and palm oil; animal oils such as beef tallow, lard and fish oil; which may be further fractionated or transesterified.

[0009] The oil phase content in the acidic oil-in-water type emulsion composition is preferably 5 to 85 % by weight, more preferably 10 to 80 % by weight, particularly preferably 20 to 80 % by weight. In particular, the oil phase in the mayonnaise-type composition is preferably 20 to 85 % by weight, more preferably 25 to 80 % by weight, particularly preferably 30 to 80 % by weight. The oil phase content in the dressing-type composition is preferably 5 to 60 % by weight, more preferably 10 to 55 % by weight, most preferably 20 to 50 % by weight.

[0010] The crystallization-inhibitor used in the present invention is preferably selected from polyglycerol fatty acid esters, sucrose fatty acid esters and sorbitan fatty acid esters. The polyglycerol fatty acid esters are preferably those in which the average polymerization degree of glycerol is 2 to 12, the number of carbon atoms in the fatty acid moiety is 12 to 22, and the degree of esterification is at least 70 %. Furthermore, the HLB of polyglycerol fatty acid esters is preferably lower than 4.5, most preferably lower than 3.5. In the present invention, the average polymerization degree (Mw) is determined by the following equation from the relationship between the polymerization degree (n) and hydroxyl value (OHV) of polyglycerol and the theoretical value.

$$\text{OHV} = 56110(n + 2)/\text{Mw}$$

$$\text{Mw} = 74n + 18$$

[0011] The sucrose fatty acid esters are preferably those in which the degree of esterification with fatty acids having 12 to 22 carbon atoms is at least 50 %, and the remaining hydroxyl groups (not esterified with above-mentioned fatty acids) are acetylated. Furthermore, the sucrose fatty acid esters preferably have an HLB lower than 3, more preferably lower than 2. The sorbitan fatty acid esters are preferably those in which the number of carbon atoms in the fatty acid moiety is 12 to 22, and the HLB is lower than 3, more preferably lower than 2.5. One or more of these crystallization inhibitors may be used, and the content thereof must be 0.5 to 5.0 % by weight, preferably 0.6 to 3.0 % by weight from the viewpoints of sufficient crystallization-

inhibiting effect at low temperatures and flavor as food. The polyglycerol fatty acid esters are particularly preferred from the viewpoint of crystallization-inhibiting effect at varied temperatures.

[0012] The water phase in the acidic oil-in-water type emulsion composition according to the present invention may contain water; vinegar; common salt; condiments such as sodium glutamate; saccharides such as sugar and starch syrup; seasonings such as sake and sweet sake; various kinds of vitamins; organic acids; spices; various kinds of vegetables or fruits; thickeners such as xanthan gum; dairy products such as milk; various kinds of fruit juices; proteins such as soybean protein; various kinds of phosphates; etc.

[0013] Yolk may be used in the acidic oil-in-water type emulsion composition according to the present invention for the purpose of emulsifying and improving flavor. Yolk may be used in any form such as raw, frozen, powdery, salted or sugared. Furthermore, it may also be used in the form of the whole egg containing egg white. The content of the yolk in the acidic oil-in-water type emulsion composition according to the present invention is preferably 5 to 20 %, more preferably 7 to 17 %, most preferably 8 to 15 % based on weight of raw yolk, from the viewpoint of improvement in flavor.

[0014] In the acidic oil-in-water type emulsion composition according to the present invention, a proportion (hereafter referred to as "lyso proportion") of lysophospholipid to total phospholipids contained is preferably at least 15 %, more preferably at least 25 %, most preferably 29 to 60 % from the viewpoints of shelf stability, appearance and flavor. A part or the whole of the lysophospholipid is preferably derived from yolk and/or soybean, most preferably from yolk. Furthermore, a part or the whole of the lysophospholipid is preferably enzyme-treated yolk. The enzyme used in the enzyme treatment of yolk is preferably esterase, lipase or phospholipase, more preferably lipase or phospholipase, most preferably phospholipase. Among various kinds of phospholipases, phospholipase A, i.e., phospholipase A1 or A2 is most preferred.

[0015] The acidic oil-in-water type emulsion composition according to the present invention preferably has a pH of 2.5 to 5.0, more preferably 3.0 to 4.5, most preferably 3.4 to 4.2.

[0016] The acidic oil-in-water type emulsion composition according to the present invention can be produced in accordance with, for example, the following process. Oil components such as DAG and the crystallization inhibitors are first mixed to prepare an oil phase. Yolk and other water-soluble raw materials are then mixed to prepare a water phase. The oil phase is added to the water phase, and the mixture is homogenized following preliminary emulsification if needed, whereby an acidic oil-in-water type emulsion composition can be obtained. Examples of homogenizers include high-pressure homogenizers such as Mountaingorin and Microfluidizer, ultrasonic emulsifiers, colloid mills, agitating homomixers, and Milder.

[0017] The average droplet diameter of oil droplets in the acidic oil-in-water type emulsion composition produced in such a manner is preferably 0.5 to 40.0  $\mu\text{m}$ , more preferably 1.0 to 15.0  $\mu\text{m}$ , most preferably 1.5 to 12.0  $\mu\text{m}$  when measured by a laser diffraction method (using a laser diffraction particle size analyzer).

[0018] When the acidic oil-in-water type emulsion composition according to the present invention is mayonnaise or dressing, it is more preferable to have the following average droplet diameter. In the case of mayonnaise, the average droplet diameter is preferably 0.5 to 20.0  $\mu\text{m}$ , more preferably 1.0 to 15.0  $\mu\text{m}$ , most preferably 1.5 to 10.0  $\mu\text{m}$ .

In the case of dressing, the average droplet diameter is preferably 0.5 to 40.0  $\mu\text{m}$ , more preferably 1.0 to 40.0  $\mu\text{m}$ , most preferably 5.0 to 40.0  $\mu\text{m}$ .

[0019] Examples of product form of the acidic oil-in-water type emulsion composition according to the present invention include those defined by JAS as dressing, semi-solid dressing, emulsified liquid dressing, mayonnaise, salad dressing and French dressing, but not limited to. All kinds of products, widely accepted as mayonnaise and dressing, correspond thereto.

[0020] In the following examples, the HLB of the crystallization inhibitors was calculated out in accordance with the Griffin's empirical equation for polyglycerol fatty acid esters, and sorbitan fatty acid esters. For sucrose fatty acid esters, emulsification method was used to determine HLB.

#### Referential Example 1:

##### Preparation of Oil Composition 1

[0021] Lipozyme IM (product of Novo Nordisk Industry Co.) was added to a mixture of rapeseed oil fatty acid (650 parts by weight) and glycerol (107 parts by weight) and subjected to esterification reaction at 40 DEG C for 5 hours under 7 hPa. The resultant reaction mixture was then subjected to molecular distillation (235 DEG C, 0.07 hPa). The distillate thus obtained was then bleached, washed with water and deodorized at 235 DEG C for 2 hours to obtain Oil Composition 1 having a composition shown in Table 1. Referential Example 2:

##### Preparation of Oil Composition 2

[0022] A mixture of soybean oil fatty acid (650 parts by weight), after the content of saturated fatty acids had been reduced by winterization, glycerol (107 parts by weight) and calcium hydroxide (2 parts by weight) was reacted at 230 DEG C for 0.5 hours under nitrogen gas atmosphere, the reaction mixture was allowed to stand for 12 hours to separate the glycerol phase, and then the resultant oil phase (oil composition) was washed with a 50 % (by weight) aqueous solution of citric acid whose proportion was 2 parts by weight to 100 parts by weight of the oil phase followed by centrifugation to afford oil composition. The resultant oil composition was then subjected to molecular distillation (235 DEG C, 0.07 hPa), and the distillate thus obtained was then bleached, washed with water and deodorized at 235 DEG C for 2 hours to obtain Oil Composition 2 having a composition shown in Table 1.

<tb><TABLE> Id=Table 1 Columns=4

<tb>

<tb>Head Col 1 to 2:

<tb>Head Col 3 to 4: Oil composition

<tb>

<tb>SubHead Col 1 to 2:

<tb>SubHead Col 3>1:

<tb>SubHead Col 4>2:

<tb>Composition of

\*<1>

oil<SEP>Triacylglycerol<SEP>13.8<SEP>13.5  
 <tb><SEP>Diacylglycerol<SEP>84.7<SEP>85.2  
 <tb><SEP>Monoacylglycerol<SEP>1.2<SEP>1.0  
 <tb><SEP>Free fatty acid<SEP>0.3<SEP>0.3  
 <tb>Fatty acid

\*<2>

composition<SEP>C16: 0<SEP>4.2<SEP>2.6  
 <tb><SEP>C18: 0<SEP>1.9<SEP>0.7  
 <tb><SEP>C18: 1<SEP>58.3<SEP>30.0  
 <tb><SEP>C18: 2<SEP>21.3<SEP>57.5  
 <tb><SEP>C18: 3<SEP>10.7<SEP>6.8  
 <tb><SEP>C20: 0<SEP>0.7<SEP>1.5  
 <tb><SEP>C20: 1<SEP>1.8<SEP>0.4  
 <tb><SEP>C22: 0<SEP>0.2<SEP>0.1  
 <tb><SEP>C22: 1<SEP>0.8<SEP>0.1

\*1: Analyzed by gas chromatography after silylation.

\*2: Analyzed by gas chromatography after methylation (Number of carbon atoms in fatty acid: number of carbon-carbon double bonds).

<tb></TABLE>

### Referential Example 3:

#### Preparation 1 of enzyme-treated yolk

[0023] A yolk solution (750 g) containing common salt (10 % by weight) was mixed with water (250 g). After the resultant mixture was preheated at 50 DEG C for 10 minutes, Phospholipase A2 (enzymatic activity: 10,000 IU/mL) was added to the mixture. The proportion of phospholipase to yolk solution was 100 ppm. Reaction was carried out for 3 to 5 hours, thereby obtaining Enzyme-treated Yolk Solution 1.

[0024] After a yolk solution (850 g) containing common salt (10 % by weight) was mixed with water (150 g), and the resultant mixture was heated to 50 DEG C, the above-described Phospholipase A2 was added to the mixture. The proportion of phospholipase to the yolk solution was 40 ppm. Reaction was carried out for 20 hours, thereby obtaining Enzyme-treated Yolk Solution 2.

[0025] After a yolk solution (850 g) containing common salt at a concentration of 10 % by weight was mixed with water (150 g), and the resultant mixture was preheated to 50 DEG C, Phospholipase A1 (enzymatic activity: 4,000 IU/mL) was added to the mixture. The proportion of phospholipase to the yolk solution was 200 ppm. Reaction was carried out for 5 hours, thereby obtaining Enzyme-treated Yolk Solution 3.

[0026] The lyso proportion was calculated out in accordance with the following method.

[0027] The reaction product was extracted repeatedly with a mixed solvent (3:1) of chloroform:methanol to obtain a lipid mixture. The resultant lipid mixture was subjected to thin layer chromatography to separate various kinds of phospholipids by two-

dimensional thin layer chromatography of the first dimension: chloroform-methanol-water (65:25:49) and the second dimension: butanol-acetic acid-water (60:20:20). The amounts of the separated phospholipids were measured by means of a commercially available measuring kit (permanganate ashing method, Phospholipid Test Wako, trade name; product of Wako Pure Chemical Industries, Ltd.) to calculate out contents of lysophospholipids and total phospholipids. The lyso proportion (%) was defined as "(total amount of phosphorus in lysophospholipid fraction/total amount of phosphorus in all phospholipid fractions) x 100".

#### Referential Example 4:

#### Preparation 2 of enzyme-treated yolk

[0028] Twice amount of water by volume was added to Enzyme-treated Yolk Solution 1 obtained in Referential Example 3 and stirred. The mixture was then spray-dried under air (inlet air temperature=170 DEG C, outlet air temperature=70 DEG C) to obtain enzyme-treated yolk powder. Examples 1 to 13 and Comparative Examples 1 to 6:

[0029] Oil phases and water phases of their corresponding compositions shown in Tables 2 and 3 were prepared in a conventional manner. After each of the oil phases was added to its corresponding water phase while stirring the water phase to preliminarily emulsify the oil phase, the emulsion was homogenized by a colloid mill (5,000 rpm, clearance: 0.35 mm) to prepare mayonnaise having an average emulsified droplet diameter of 2.5 to 3.5  $\mu$ m. The mayonnaise was charged into a mayonnaise tube (100 g). In respect to Example 11 and Comparative Example 4, each mayonnaise was prepared having the average droplet diameter of 8.1 to 9.5 and then charged into a mayonnaise tube (100g)

[0030] Mayonnaise samples thus obtained were stored for 1 month to test under low temperature (-5, 0 and 5 DEG C) and programmed temperature (max 25 DEG C, min 0 DEG C) and then left to stand at room temperature for 3 hours. Thereafter, the appearances and physical properties thereof were evaluated by 6 panels in accordance with the following evaluation standards. The average values of these evaluations are shown in Table 2.

#### Evaluation standards:

##### (1) Appearance:

[0031] The appearance of each mayonnaise sample charged into a tube was visually evaluated.

3: Good;

2: Signs of separation was observed;

1: Obvious separation has taken place.

## (2) Physical property

[0032] The mayonnaise sample squeezed out of the tube was visually evaluated

3: Good;

2: Signs of roughening on the surface and/or water separation was observed;

1: Obvious roughening on surface and/or water separation has taken place.

EMI14.1

EMI15.1

EMI16.1

[0033] Comparative Examples 1 and 2 relate to mayonnaise devoid of crystallization inhibitors, and both mayonnaise samples underwent demulsification (oil-off) after storage for 1 month at -5 DEG C and 0 DEG C and caused separation, surface roughness and water separation. Comparative Example 3 relates to mayonnaise in which excessive amount of polyglycerol fatty acid ester (0.4 % by weight) was used in the oil phase. Sufficient emulsion stability was unable to be achieved in this mayonnaise

[0034] On the other hand, when a proper amount of polyglycerol fatty acid ester (Examples 1 to 3 and 6 to 8), sorbitan fatty acid ester (Example 4) or sucrose fatty acid ester (Example 5) was used as crystallization inhibitor, no demulsification was observed even after storage for 1 month at -5 DEG C to 5 DEG C, and both appearance and physical property were good.

[0035] Emulsion stability tends to be improved when the ratio of oil phase is lowered, but sufficient improvement cannot be obtained in the absence of crystallization inhibitor. When used in a proper amount, the crystallization inhibitor was able to bring sufficient improvement in the emulsion stability (Examples 11 to 13 and Comparative Examples 4 to 6).

[0036] Furthermore, the polyglycerol fatty acid ester (Examples 1 to 3 and 6 to 8) was confirmed to be the best use as a crystallization inhibitor agent both in appearance and physical property, from results of the tests under programmed temperature.

Examples 14 to 16:

[0037] French dressing (Example 14), Thousand Island dressing (Example 15) and sesame dressing (Example 16) were produced with their corresponding compositions shown in Table 4. More specifically, raw materials for an oil phase were added dropwise to a water phase under stirring to conduct preliminary emulsification. This emulsion was homogenized by a homomixer to obtain the respective dressing samples having an average emulsified droplet diameter of 4 to 12  $\mu$ m. With respect to the respective dressing samples, the same evaluation as in Example 1 was conducted by 6 panelists. As a result, all the dressing samples were good in both appearance and physical property even after storage for 1 month at -5 DEG C to 5 DEG C as shown in Table 4.

## EMI19.1

## Industrial Applicability

[0038] As described above, the acidic oil-in-water type emulsion compositions according to the present invention are so excellent in shelf stability at low temperatures that they do not crack during storage at a low temperature in a refrigerator though they contain DAG at a high concentration, also good in appearance and flavor and useful as diets or lipid metabolism-improving foods.

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